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A MODIFIED NO_x SAMPLER FOR DETECTION OF HYDROGEN CHLORIDE IN AMBIENT AIR

ANTHONY M. WACHINSKI

HQ USAFA/DFCE
USAF ACADEMY, DEPT OF CIVIL ENGINEERING
COLORADO SPRINGS CO 80840-5841

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PREFACE

This research was conducted by the Department of Civil Engineering, United States Air Force Academy, CO. It was conducted under Project Order 84-37, and JON 19009014 for the Headquarters Air Force Engineering and Services Center, Engineering and Services Laboratory (AFESC/RDVS), Tyndall AFB FL 32403. Maj Anthony M. Wachinski was the USAFA Principal Investigator, and Lt Glenn D. Seitchek was the AFESC Project Officer.

The work began in January, 1984 and was completed in June, 1985. This report covers the results of the literature review and feasibility analysis of developing a hydrogen chloride (HCl) measurement device.

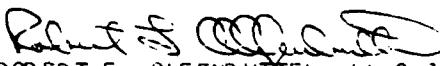
The authors wish to thank Robert E. Treybal and the McGraw-Hill Book Company for copyright permission to use Figure 2.3, "Collision Function for Diffusion" from the book Mass Transfer Operations. This figure appears as Figure 1 in this report.

This report has been reviewed by the Public Affairs Office and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nationals.

This report has been reviewed and is approved for publication.


GLENN D. SEITCHEK, 1Lt, USAF
Project Officer


KENNETH T. DENBLEYKER, Maj, USAF
Ch, Environmental Sciences Branch


ROBERT F. OLFENBUTTEL, Lt Col, USAF, BSC
Chief, Environics Division

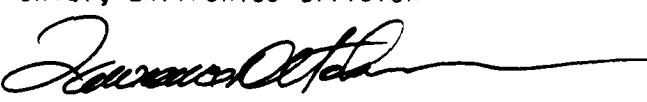

LAWRENCE D. HOKANSON, Colonel, USAF
Director, Engineering and Services
Laboratory

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SECTION I

INTRODUCTION

A. OBJECTIVE:

The objective of this project was to develop a hydrogen chloride-measuring device that was portable and lightweight, (i.e., would weigh less than 10 pounds), be power-independent, (using only low-power direct current if necessary), be low cost (less than \$100), reuseable, and provide some degree of quantification, (i.e., numbers greater or less than).

B. BACKGROUND

Some emissions of environmental concern are produced during the launch of the Space Shuttle, Titan missiles, and other projectiles employing similar solid rocket motors (SRMs). Of particular importance are the SRMs, which use ammonium perchlorate as an oxidizer, and powdered aluminum as a fuel and burn rate stabilizer. The exhaust products produced from the fuel include aluminum oxide (Al_2O_3), hydrogen chloride (HCl), and water vapor (H_2O). Of particular interest is HCl.

To effectively evaluate the impact of hydrogen chloride on the surrounding environment, ground-level concentrations occurring in an array downwind from the launch must be known. Mapping these concentrations is complicated by the large land areas to be monitored, complex terrain, and lack of electrical power. As such, the use of bubblers (liquid impingers) is prohibitive because these require electrical power for operation.

C. SCOPE/APPROACH:

A review of the literature culminated in the investigation of three possible passive techniques to measure HCl under conditions outlined in the Statement of Work. These detectors included a passive dosimeter, using molecular diffusion to quantify the HCl and air transferred through a tube to an absorbent containing sodium hydroxide during a given exposure time (Reference 1); a dosimeter manufactured by GMD Systems, Inc.* which uses Henry's Law to establish equilibrium between the HCl vapor and a liquid medium (distilled water); and a piezoelectric quartz crystal detector as a microbalance, using triphenylamine and trimethylamine-hydrochloride (Reference 2) enabling detection of hydrogen chloride at part per billion levels.

Analytical methods for measuring hydrogen chloride in ambient air are summarized by both Gregory (Reference 3) and Swoboda and Naugle (Reference 4). The techniques discussed include bubbler or impinger, coulometry, chemiluminescence, pH indicators, dosimeter tubes using an air pump, copper plates, mineral oil dishes, electrets, impingers, proprietary chromatography

* Gas Monitoring Dosimeters Systems, Inc. (GMD, Inc.), 345 Thomas Road, McMurray, PA,

tubes, charcoal tubes, sophisticated particulate-measuring systems, and biological monitoring devices. Readers are directed to Gregory (Reference 3) and Swoboda and Naugle (Reference 4) for a detailed discussion of each technique. Coated piezoelectric quartz crystals have not been used in a field environment as many problems require solving (Reference 5). The passive dosimeter has been used successfully to measure nitrogen dioxide (NO_2) as a personal sampler in the workplace (Reference 6). The GMD, Inc. dosimeter has been used to measure monomethyl hydrazine (MMH) with limitations inherent to this type of sampler.

The molecular diffusion sampler, otherwise known as the Palms tube, is discussed in this report. (Reference 1)

SECTION II
PALMES TUBE

A. BACKGROUND

1. Theory

The personal sampler proposed by Palmes and Gunnison (Reference 1) uses the principle of molecular gas diffusion derived from Ficks first Law, i.e., the unidirectional, isothermal diffusion of gas G_1 through a mixture of gases at constant pressure can be expressed in terms of Equation (1):

$$J_1 = -D_{12} \left(\frac{dc_1}{dx} \right) \quad (1)$$

Where J_1 = molal flux of species 1 ((moles/cm²)/sec)

D_{12} = diffusion coefficient of gas 1 through gas 2(cm²/sec)

$\frac{dc_1}{dx}$ = concentration gradient ((moles/cm³)/cm),

c_1 = concentration of diffusing gas G_1 in moles /cm³

x = distance in direction of diffusion (cm)

The right hand side of the equation is negative since the flux is in the direction of decreasing concentration.

If the diffusion chamber is a flat-bottomed cylinder having a reagent trap at its base, the concentration of G_1 at the base can be assumed to be zero. Further, if the other end of the chamber (or tube) is exposed to the ambient air to be sampled, then

When $x = L$ (Length of the sampling tube)

$c = 0$, i.e., the concentration of G_1 at the base of the tube is zero, as the gas is completely absorbed by the reagent trap.

When $x = 0$ (the open-end of the tube exposed to ambient air)

$c_1 = C_0$ (the concentration of G_1 in the atmosphere)

If the cross-sectional area of the tube is A (cm²) and the time the tube is exposed to the atmosphere is t (sec), the number of moles of G_1 which diffuses through the tube is given by the equation

$$Q = JAt = \frac{-D_{12} c_1 A t}{L} \quad (2)$$

Knowing the number of moles of G_1 collected, the area and length of the collecting tube, the collection time, and diffusivity of gas G_1 through

ambient air, the concentration of gas G_1 can be determined and the device can be used as a quantitative sampler.

As the dimensions of the sampling tube and the time of exposure are readily determined, the diffusion coefficient of the gas to be collected must be determined. Many methods are available to determine this parameter.

Hirschfelder, et al. (Reference 7) have summarized the following method for determining the diffusivity

$$D_{AB} = \frac{0.0009292}{P} T^{3/2} \left(\frac{1}{M_A} + \frac{1}{M_B} \right)^{1/2} r_{AB}^2 [f(KT/\epsilon_{AB})] \quad (3)$$

Where:

D_{AB} = diffusion coefficient of Gas Species A through Gas Species B (cm^2/sec)

T = absolute temperature, Degrees K

M_A, M_B = molecular weights of A and B, respectively

P = absolute pressure, atmospheres

r_{AB} = molecular separation at collision, angstroms

$$= \frac{(r_A + r_B)}{2}$$

ϵ_{AB} = energy of molecular interaction, ergs

$$= \sqrt{\epsilon_A \epsilon_B}$$

K = Boltzmann Constant = 1.38×10^{-16} erg/ $^\circ\text{K}$

$f(KT/\epsilon_{AB})$ = a collision function given by Figure 1

For a hydrogen chloride and air mixture, the following table summarizes the constants.

TABLE 1. FORCE CONSTANTS OF GASES (References 7,8)

<u>Gas</u>	<u>$\epsilon/K(^{\circ}K)$</u>	<u>$r(\text{Å})$</u>	<u>M</u>
Air	97	3.617	28.96
HCl	350	3.305	36.5

Another method of evaluating diffusivity at moderate temperatures and pressures is the Chapman-Enskog Formula (Reference 10).

$$D_{AB} = \frac{0.0018583 T^{3/2}}{P} \frac{(1/M_A + 1/M_B)^{1/2}}{\sigma_{AB}^2 \Omega_{AB}} \quad (4)$$

Where,

D_{AB} = bulk diffusivity

T = temperature, degrees K

M_A, M_B = molecular weights of gases A and B

P = total pressure of the gas mixture, atm

σ_{AB} = Constants in the Lennard-Jones potential-energy function for the molecular pair.

Ω_{AB} = Collision integral, which would be unity if the molecules were rigid spheres, a function KT/ϵ for real gases (Table 2).

K = Boltzmann's constant.

Since the Lennard-Jones potential-energy function is used the equation is strictly valid only for nonpolar gases (Reference 10). The equation is most satisfactory for mixtures of nonpolar gases or for a polar with a nonpolar gas (Reference 9).

TABLE 2. COLLISION INTEGRAL FOR LENNARD-JONES POTENTIAL
(Adapted from Reference 12, p. 366)

<u>KT/ϵ</u>	<u>Ω_{AB}</u>
1.35	1.253
1.40	1.233
1.45	1.215
1.50	1.198
1.55	1.182
1.60	1.167
1.65	1.153

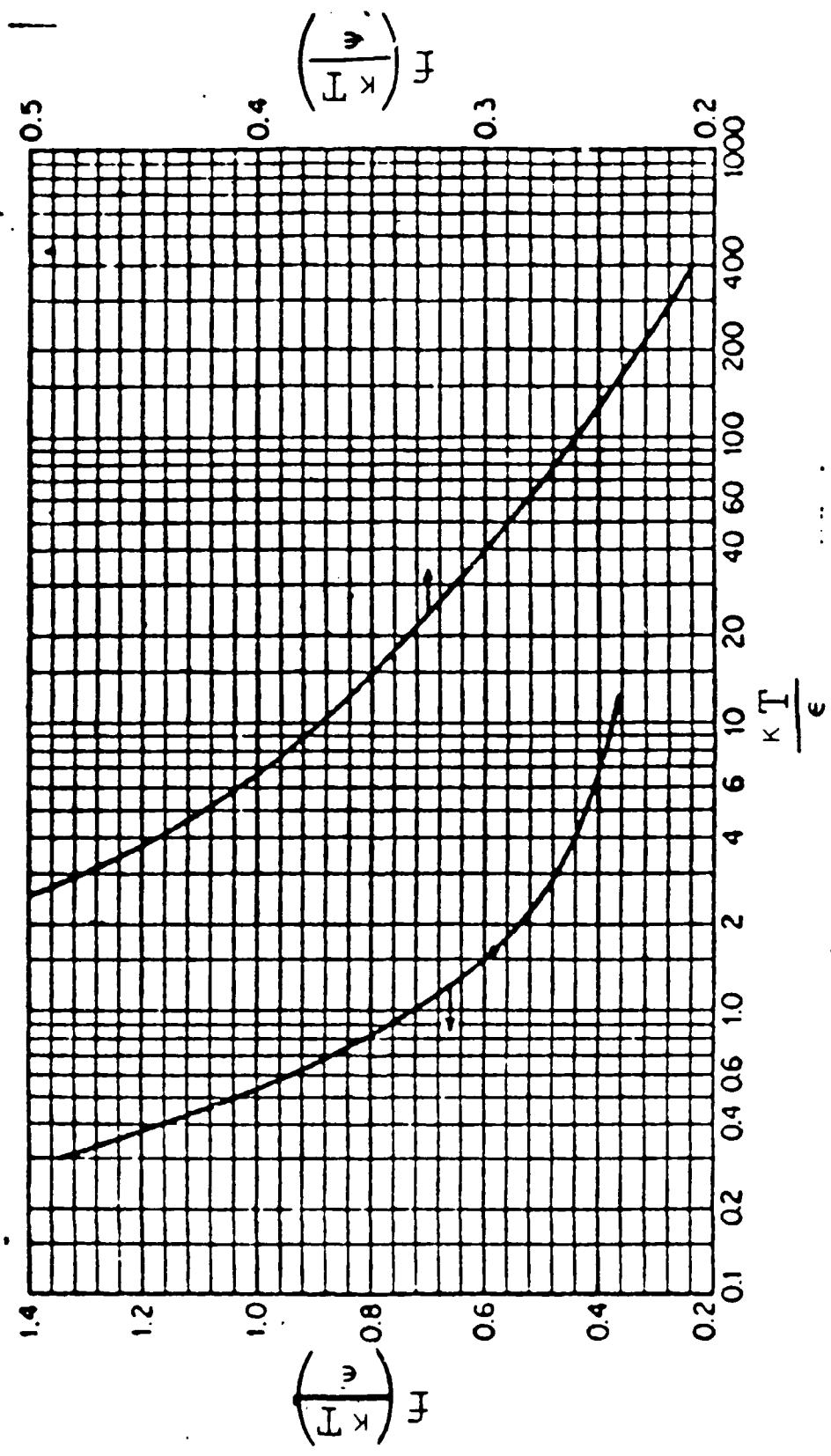


Figure 1. Collision Function for Diffusion (courtesy McGraw-Hill)

2. Example

a. Hirschfelder-Method. Estimate the diffusity of HCl through air at 1 atmosphere, 70 degrees F, using the method proposed by Hirschfelder, Bird, and Spatz (Reference 7).

$$T = 294.1 \text{ degrees K}$$

$$P = 1 \text{ atm}$$

$$M_A = 36.5 \quad (\text{Table 1})$$

$$M_B = 28.96 \quad (\text{Table 1})$$

$$r_A = 3.305 \text{ angstroms} \quad (\text{Table 1})$$

$$r_B = 3.617 \text{ angstroms} \quad (\text{Table 1})$$

$$r_{AB} = \frac{3.305 + 3.617}{2} = 3.461 \text{ angstroms}$$

$$\epsilon_{A/K} = 360 \text{ degrees K} \quad (\text{Table 1})$$

$$\epsilon_{B/K} = 97 \text{ degrees K} \quad (\text{Table 1})$$

$$\epsilon_{AB/K} = \sqrt{\frac{\epsilon_A \epsilon_B}{K^2}} = \sqrt{(360)(97)} = 186.87 \text{ degrees K}$$

$$\frac{KT}{\epsilon_{AB}} = \frac{294.1}{186.9} = 1.57$$

$$f\left(\frac{KT}{\epsilon_{AB}}\right) = 0.59 \quad (\text{Figure 1})$$

$$D_{AB} = \frac{(0.0009292)(294.1)^{3/2}}{(1)(3.461)^2(0.59)} \left(\frac{1}{36.5} + \frac{1}{28.96} \right)^{1/2}$$

$$D_{AB} = \frac{(0.0009292)(5043.6)(0.249)}{(1)(11.979)(0.59)}$$

$$D_{AB} = 0.165 \text{ cm}^2/\text{s}$$

b. Chapman-Enskog Formula. Calculating the bulk diffusity for the conditions in the previous example using the Chapman-Enskog Formula:

For Air

$$\frac{\epsilon_B}{K} = 97 \text{ degrees K} \quad r_B = 3.617 \text{ angstroms}$$

For HCl

$$\frac{\epsilon_A}{K} = 360 \text{ degrees K} \quad \sigma_A = 3.305 \text{ angstroms}$$

$$\sigma_{AB} = \frac{3.617 + 3.305}{2} = 3.461 \text{ angstroms}$$

Interpolating from Table 2:

$$\Omega_{AB} = 1.178$$

$$D_{AB} = \frac{0.0018583 T^{3/2} (1/M_A + 1/M_B)^{1/2}}{P^2 \sigma_{AB}^2 \Omega_{AB}} \quad (4)$$

$$D = \frac{(0.0018583) (294.1)^{3/2} \left(\frac{1}{36.5} + \frac{1}{28.96} \right)^{1/2}}{(1) (3.461)^2 (1.178)}$$

$$D = 0.165 \text{ cm}^2/\text{s}$$

B. THE SAMPLER

1. Sampler Construction:

The HCl monitoring device chosen for consideration was designed by Palmes, et al. (References 1 and 6). The HCl sampler operates on the same principle as the NO_x personal sampler except that filter paper soaked in sodium hydroxide (NaOH) is used to collect the HCl. The filter paper replaces the stainless steel screens coated with triethanolamine (TEA) used to collect NO_x in the Palmes Tube.*

A schematic of the NO_x sampler is shown in Figure 2. Photographs of the sampler are shown in Figure 3.

*The tubes were purchased from MDA Scientific, Inc, 1815 Elmdale Avenue, Glenview, Illinois 60025. The tubes are listed as a NO₂-NO_x sampler.

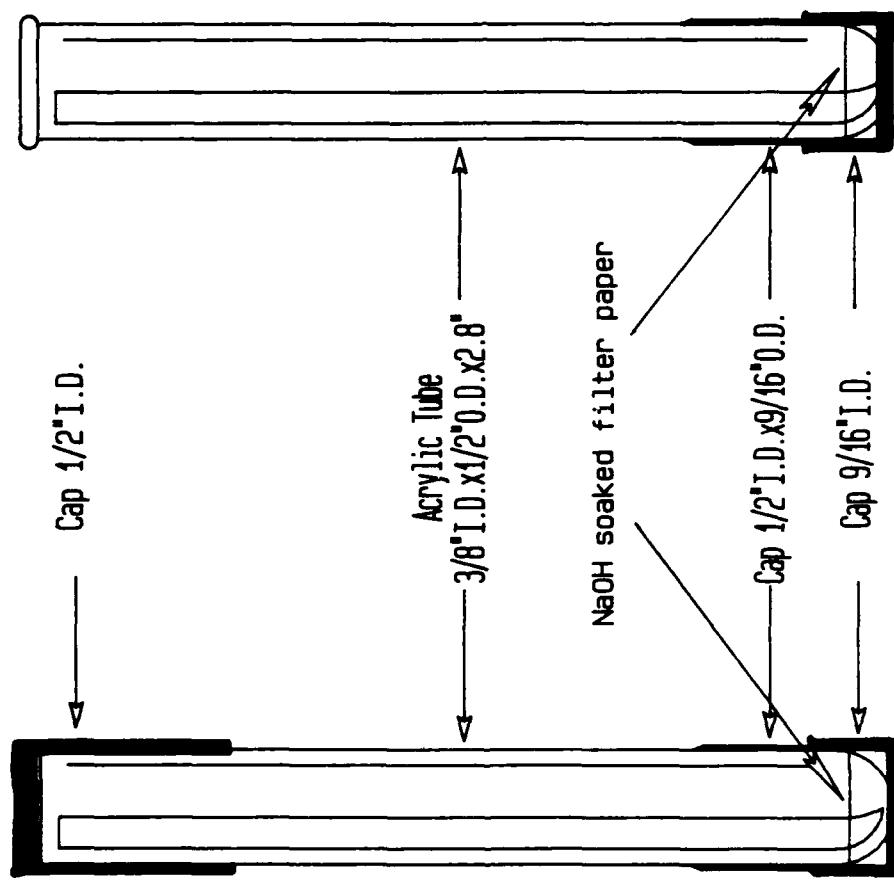


FIGURE 2. SCHEMATIC DRAWING OF HCL SAMPLER

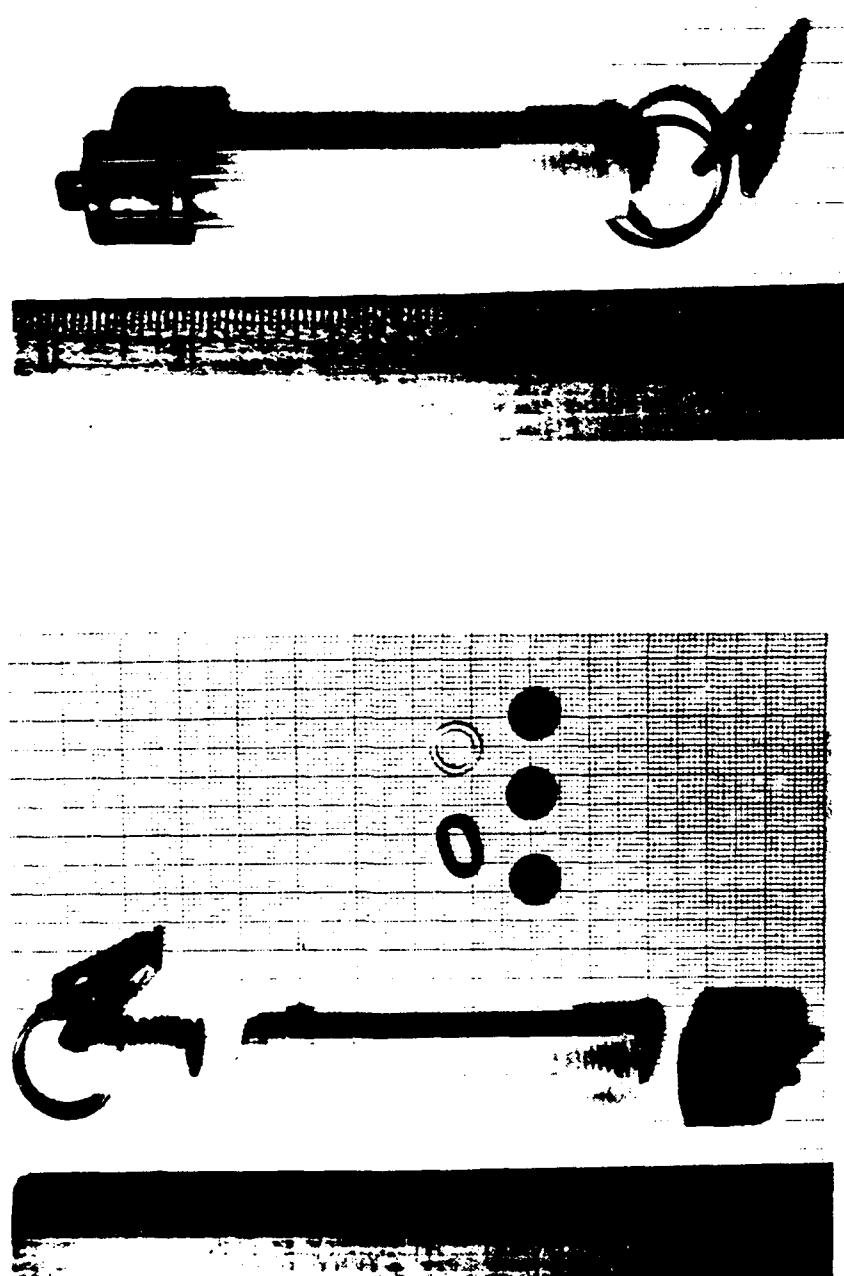


Figure 3. Photographs of HCl Tube Sampler

This design (Reference 11) uses a polypropylene tube 0.95 cm (3/8 inch) inside diameter, 1.3 cm (1/2 inch) outside diameter, with a cross-sectional area to length ratio (A/L) of 0.1 cm. The TEA-coated screens were replaced with NaOH-soaked filter paper (see Figure 4) for the HCl sampler.

An existing unit was selected to save development time. It was tested in the laboratory where data were compared to a bubbler and in a field situation at White Sands Missile Range, New Mexico.

2. Modified Sampler Design

The original design requirements were for periods ranging from 25 minutes to 24 hours. The HCl sampler design meets these requirements. However, if the dosimeters are to be used in the outlying or far field area, a new design is required, as HCl concentrations of 5-10 ppm for the time periods of 20-30 seconds can be expected. The following describes a new design to meet sampling requirements for 10 ppm concentration for 30 seconds.

Design Parameters:

$$T = 294.1 \text{ degrees K}$$

$$D = 0.165 \text{ cm}^2/\text{s}$$

$$t = 30\text{s}$$

$$C_0 = 10 \text{ ppm}$$

$$Q = 1.0 \text{ microgram (sensitivity of chloride detection method)}$$

Find: $A(\text{cm}^2)$; $L(\text{cm})$

$$Q = D \left(\frac{A}{L} \right) C_0 t$$

$$\frac{A}{L} = \frac{Q}{D C_0 t}$$

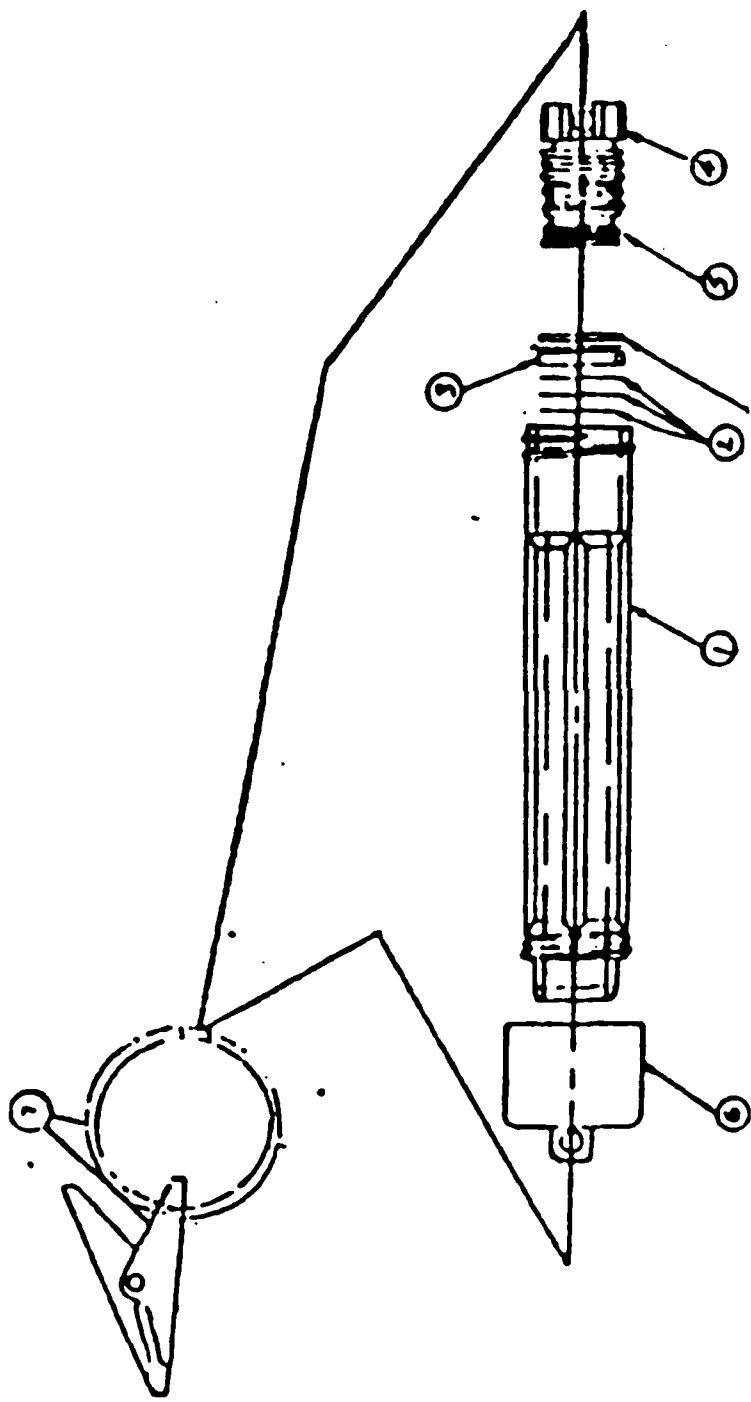
Change Q to moles:

$$\frac{1 \times 10^{-6} \text{ grams}}{35.453 \text{ grams/mole}} = 28.21 \times 10^{-9} \text{ moles}$$

Change ppm to moles/cm³, one mole of gas occupies a volume of 22.41 liters at standard temperature and pressure.

$$\frac{V_1}{T_1} = \frac{V_2}{T_2}$$

$$V_2 = \frac{T_2}{T_1} V_1 = \frac{(294.1)(22.41)}{(273)}$$



1. POLYPROPYLENE SAMPLING TUBE
2. NaOH SOAKED FILTER PAPER
3. POLYETHYLENE SPACE
4. SCREW IN CAP
5. VITON O-RING
6. PROTECTIVE CAP
7. CLIP ASSEMBLY

Figure 4. Parts Description of HCl Tube Sampler

$$V_2 = 24.13 \text{ liters}$$

$$1 \text{ ppm} = \frac{1 \text{ mole}}{10^6 \text{ moles}} = \frac{10^{-6} \text{ moles}}{1 \text{ moles} \times (24.13 \text{ l/mole}) \times (1000 \text{ cm}^3/\text{l})}$$

$$= \frac{1 \times 10^{-6} \text{ moles}}{2.413 \times 10^4 \text{ cm}^3}$$

$$= 0.41 \times 10^{-10} \text{ moles/cm}^3$$

$$10 \text{ ppm} = 0.41 \times 10^{-9} \text{ moles/cm}^3$$

Substituting:

$$\frac{A}{L} = \frac{2.821 \times 10^{-10} \text{ moles}}{(0.165 \text{ cm}^2/\text{sec}) (0.41 \times 10^{-9} \text{ moles/cm}^3) (30 \text{ sec})}$$

$$\frac{A}{L} = 13.9 \text{ cm}$$

$$\underline{A = 13.9 \text{ L}}$$

Area using: 12.5 cm diameter filter paper = 122.72 cm² yields L = 8.8cm
11.0 cm diameter filter paper = 95 cm² yields L = 6.8 cm

Use the 11.0 cm diameter paper

A design to sample for 5 ppm HCl, 30-second duration (150 ppm-sec), 70 degrees Fahrenheit ambient air temperature, sensitivity of 1.0 microgram would require 15 cm diameter filter paper, area = 176.7 cm² and a length of 6.4 cm.

A schematic of a proposed design is shown in Figure 5.

Another possible configuration would involve bundling the Palms Tubes together in groups of five, seven, or nine until an acceptable area is achieved.

Two things need to be considered in the two configurations proposed above. First, for the configuration with the high A/L, any turbulence may invalidate the "still air" diffusion equation (Equation (1)). Second, for the bundled configuration to be successful, the volume of water used to wash all of the filters must be kept reasonably close to the volume used to wash a single filter in order to gain the added sensitivity.

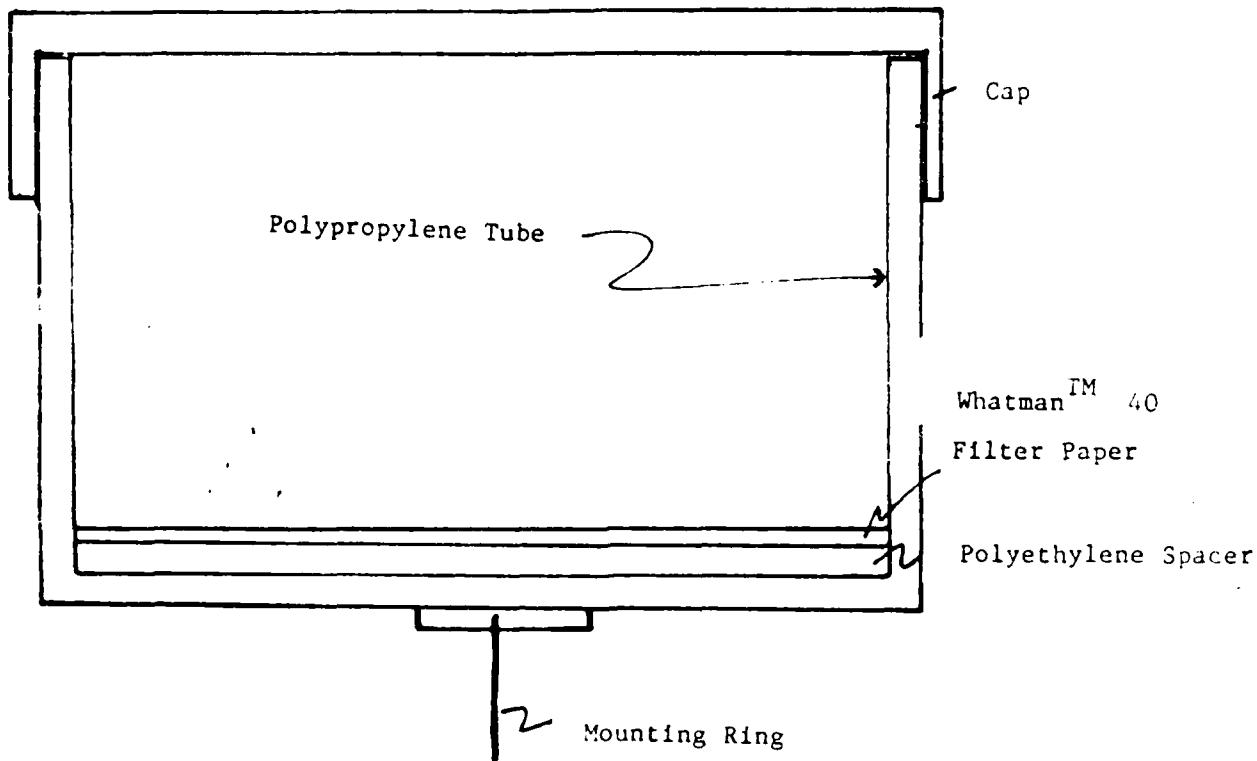


Figure 5. Proposed Design: HCl Sampler

3. Testing and Evaluation

a. Laboratory Analysis. This report summarizes the limited testing conducted at the USAF Academy. Three runs were attempted, with one yielding results. The purpose was to compare the results from the passive Palms tube system to those of an active system (bubbler).

The HCl Sampler was tested against a bubbler sampler in plastic-bag chamber by drawing a known volume of HCl and air through the chamber. The HCl sampler was attached inside the plastic bag. The bubblers were positioned outside the bag, as shown in Figure 6.

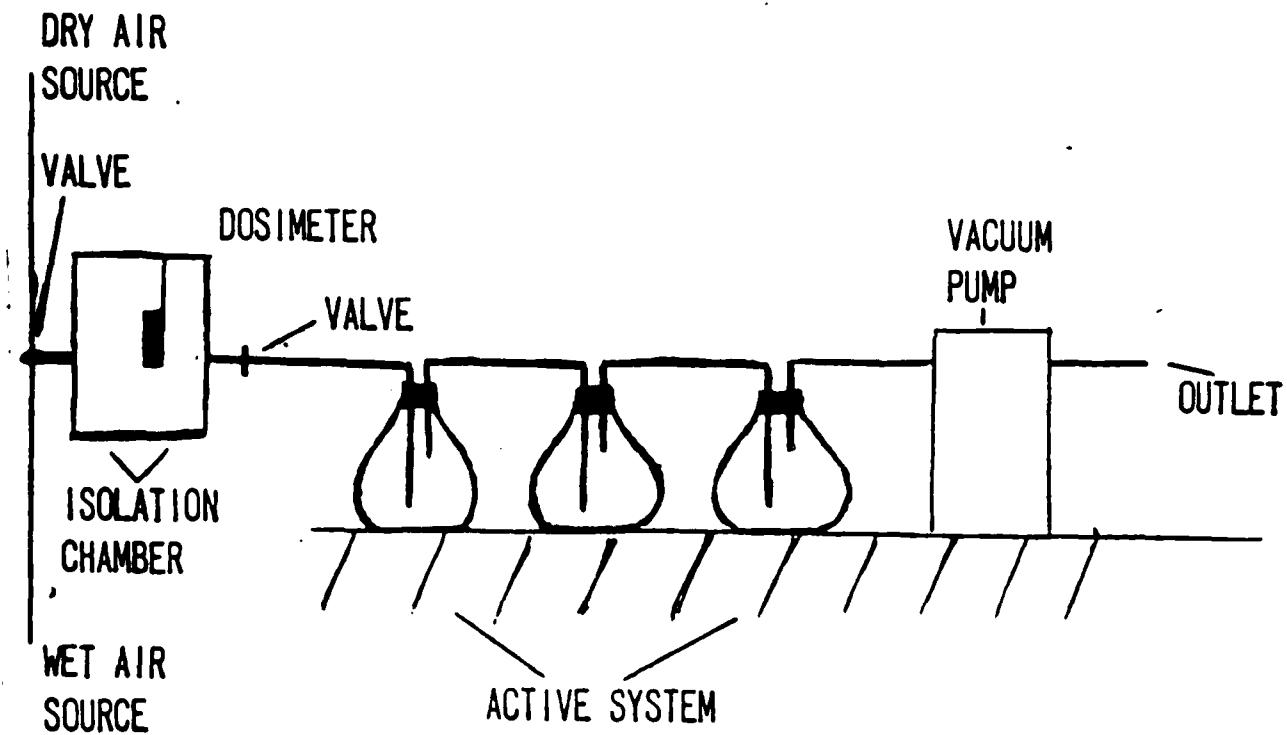


Figure 6. Experimental Apparatus

As this testing was performed by students, nonconventional methods were used, e.g., flow rates were determined using soap bubbles through a burette instead of from a rotometer. Due to USAF Academy safety restrictions, bottled HCl gas could not be used. Instead, it was generated by reacting sodium chloride (NaCl) with sulfuric acid (H₂SO₄). The amount of NaCl and H₂SO₄ decreased with time, therefore, the concentration of HCl gas in the bubbler varied with time. The active system (bubbler) consisted of two 250-milliliter Erlenmeyer flasks with associated stoppers and corks. Air was dried and cleaned before entering the system. The bubbler flow rate was 30 mL/minute.

Sodium chloride was extracted from the filter paper by letting the paper soak in 20 ml of deionized water. Chloride concentrations in both systems were measured, by a potentiometric titration using a 1.0 M silver nitrate solution and a glass and silver -silver chloride electrode system. Three runs were planned, although only one was performed because of analytical problems.

b. Results and Discussion

Results of the first run were as follows:

HCl Sampler

$Q = 2.82 \times 10^{-4}$ moles of hydrogen chloride

$C = 1.21 \times 10^{-5}$ moles/cm³ of HCl (Average concentration using total volume of air which flowed through the chamber)

Stoichiometric Value

$C = 4.41 \times 10^{-6}$ moles/cm³ of HCl (Average concentration assuming total depletion of NaCl and using total volume of air which flowed through the chamber)

HCl Bubbler

$C = 4.37 \times 10^{-6}$ moles/cm³ of HCl (Average concentration using total volume of air which flowed through the chamber)

A lack of a clear equivalence point was noted by the students during the analysis, i.e., unstable readings after addition of the silver nitrate (AgNO_3). Probable causes include the high concentration of AgNO_3 used as the titrant and the lack of experience of the students. These factors may have contributed to the HCl sampler concentration being about three times greater than the theoretical maximum concentration.

Field tests were conducted at the White Sands Missile Range (WSMR) using actual rocket exhaust. These results will be published in a WSMR technical report.

The NaOH treated filter, which was exposed to HCl , was washed with 2 ml of water and analyzed on a Dionex Model 2020I Ion Chromatograph. The sensitivity of this method is about 1.0 micrograms of Cl^- , thus, the detection limit of the Palmes Tube, coupled with this detection method, is about 3.9×10^4 ppm sec. This would be useful only for very high concentrations of HCl or smaller concentrations sustained over a long period of time.

SECTION III

CONCLUSION AND RECOMMENDATIONS

The proposed Palms tube, HCl sampler offers a viable means of sampling for HCl. The sampler will work as a qualitative device. Further work is required to determine its quantitative capabilities.

The equation proposed by Hirschfelder et al. (Reference 7) and Chapman-Enskog formula (Reference 9) gave similar numbers for the bulk diffusivity of HCl in air.

Experimental values for the bulk diffusivity of HCl in air are needed.

Due to limitations placed on the students, the results of the laboratory analysis cannot be considered accurate. Therefore, further laboratory and field testing of the sampler are needed.

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